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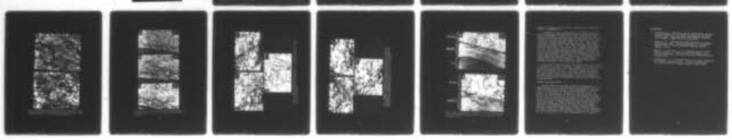

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NRL Memorandum Report 3535

NRL Engineering Materials and Chemistry Divisions Studies of the A2 Tube Failure

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June 1977

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This report discusses the hypotehsis and supporting evidence concerning the probable major failure mode of A2 microwave tubes. Such failure occurred due to loss of adequate vacuum. It was hypothesized that cracking due to tube wall (Al_2O_3)-metal electrode stresses could cause cracks to connect pores in the Al_2O_3 to the tube interior and thus vent gas in the pores left from processing into the tube. Calculations, microstructure, and fractographic evidence from this program are reported supporting this pore mechanism. Supporting pore outgassing, stress cracking and acoustic emission evidence from other studies is also noted. Limited tensile test trials of the tubes are also reported.

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FOREWORD

In January 1974, George Hass invited Roy Rice and Lynn Jarvis to join him, Henry Gray and Richard Thomas to discuss our possible interaction in a tube failure study they were involved in with Harry Diamond Laboratories. At this meeting, the nature of the A2 and related problems were presented, and a free ranging discussion was held on possible sources of gas to raise the tube vacuum levels above those required for necessary tube operation. Amongst several suggestions of possible gas desorption or diffusion, Rice noted that it should contain gases and that if stresses, especially joint stresses, were high enough, cracking would occur to connect pores to the interior of the tube. Such connection would thus allow gases in pores to vent into the tube. The investigation of this mechanism became the focus of this study with Steve Freiman supervising most of the tensile testing, Bob Jones doing most of the scanning microscopy, and Roy Rice the microstructural analysis. Throughout the study, George Hass acted as coordinator both for his program and the complete NRL program to Harry Diamond Laboratories.

SUMMARY REPORT

NRL ENGINEERING MATERIALS AND CHEMISTRY DIVISIONS STUDIES OF THE A2 TUBE FAILURE

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Engineering Materials Division

and

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INTRODUCTION

The A2 tube (see Fig. 1) problem has recently been outlined by Viechnicki.[1] As noted in the Foreword to this report, the focus of this study was to determine whether linking of pores to the tube interior by cracking was a plausible mechanism of raising A2 tube vacuum pressures to failure levels. This work and other studies show that this pore-crack mechanism is indeed a highly probable cause of tube failure, e.g. see Viechnicki.[1]

Preliminary calculations based on the small tube free internal volume (~ 0.4 cm^3), the expected porosity (3-7%), and pore size (from a few microns to several tens of microns) showed that cracks of limited size could indeed raise tube pressures to failure levels (10^{-3} - 10^{-4} torr) if the pores contained gas at about atmospheric pressure as expected. In fact, this could still be a failure mechanism even if gas pressures in pores were 1 or 2 orders of magnitude lower. It was subsequently learned that various Army investigations had observed cracks in the vicinity of the metal - Al_2O_3 seals (Fig. 1), e.g. see Viechnicki.[1] Further, calculations[2] indicated that substantial, e.g. 30,000 psi tensile stresses could exist in the Al_2O_3 near the braze due to metal-braze-Al_2O_3 property differences (especially thermal expansion) to cause cracks of the type observed. Thus, the primary tasks to verify this mechanism of failure were to first determine the amount of porosity and associated gas, then to verify that cracking occurred from the inside of the tube propagating outward.

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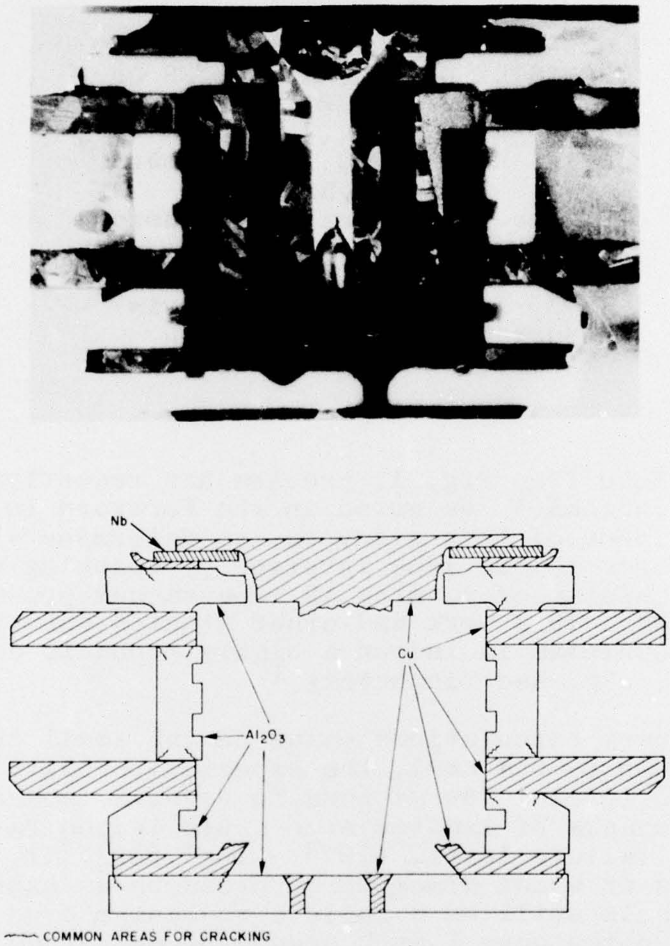

Fig. 1 — A-2 cross section. The upper portion is a photo of an actual cross section of an A-2 tube (courtesy of Dr. D. Viechnicki of AMMRC). The lower portion is a sketch identifying the materials and many of the typical likely areas for cracking.

This report presents evidence that there is indeed sufficient porosity and that many cracks do propagate in the necessary direction, i.e. from the inside-outward. Tensile tests which were conducted to determine tube failure stresses are also reported. While funding did not permit full development of these tests, it should be noted that it was felt that they could be used as an ultimate verification of the proposed crack-pore mechanisms. Thus, if cracks grew under stress (in practice from temperature fluctuations and resultant expansion and contraction) such growth under tension could be detected. Both acoustic emission and operation of the tube to act essentially as a vacuum gauge (with George Hass and colleagues) were viewed as a means of detecting such cracking. The brief tensile test trials are reported first, followed by the more extensive fractography for microstructural and crack direction analysis.

EXPERIMENTAL RESULTS*

Tensile Tests.

The approach to tensile testing was to bond a brass rod to each tube end (Fig. 2) Each unbonded end of the brass rods had a threaded recess into which an eyebolt was screwed. Each of these eyebolts was attached to an eyebolt in the test machine load train to effectively provide universal joints on both ends of tube (Fig. 2). Further, hemispherical air bearings were in the top and bottom halves of the load train to minimize parasitic stresses. Initial trails with Eastman 910 as the adhesive bonding the eyebolt to the tubes were unsuccessful, so all subsequent work was with epoxy based adhesives.

Limited trials of different epoxy systems gave scattered results which are summarized in Table 1 showing both the stress sustained in the tube Al_2O_3 wall and in the epoxy adhesive. Failure of the tube-brass bond typically occurred at or near the brass-epoxy interface. Thus, subsequent coarse knurling of the brass surface to be bonded tended to increase the stress that the adhesive joints could sustain (e.g. 50% or more). While some "bad" tubes were successfully fractured (Table 1), stresses in Al_2O_3 walls and braze joints were well below those expected for general failure, i.e. a tensile strength of 20,000-50,000 psi for the Al_2O_3. Also, no tubes designated as "good" were broken. Thus, even if stresses in or near the brazes reduced the failure stress by

*Note that except for a few Mictron tubes included in the tensile test trails, all work was on G. E. tubes.

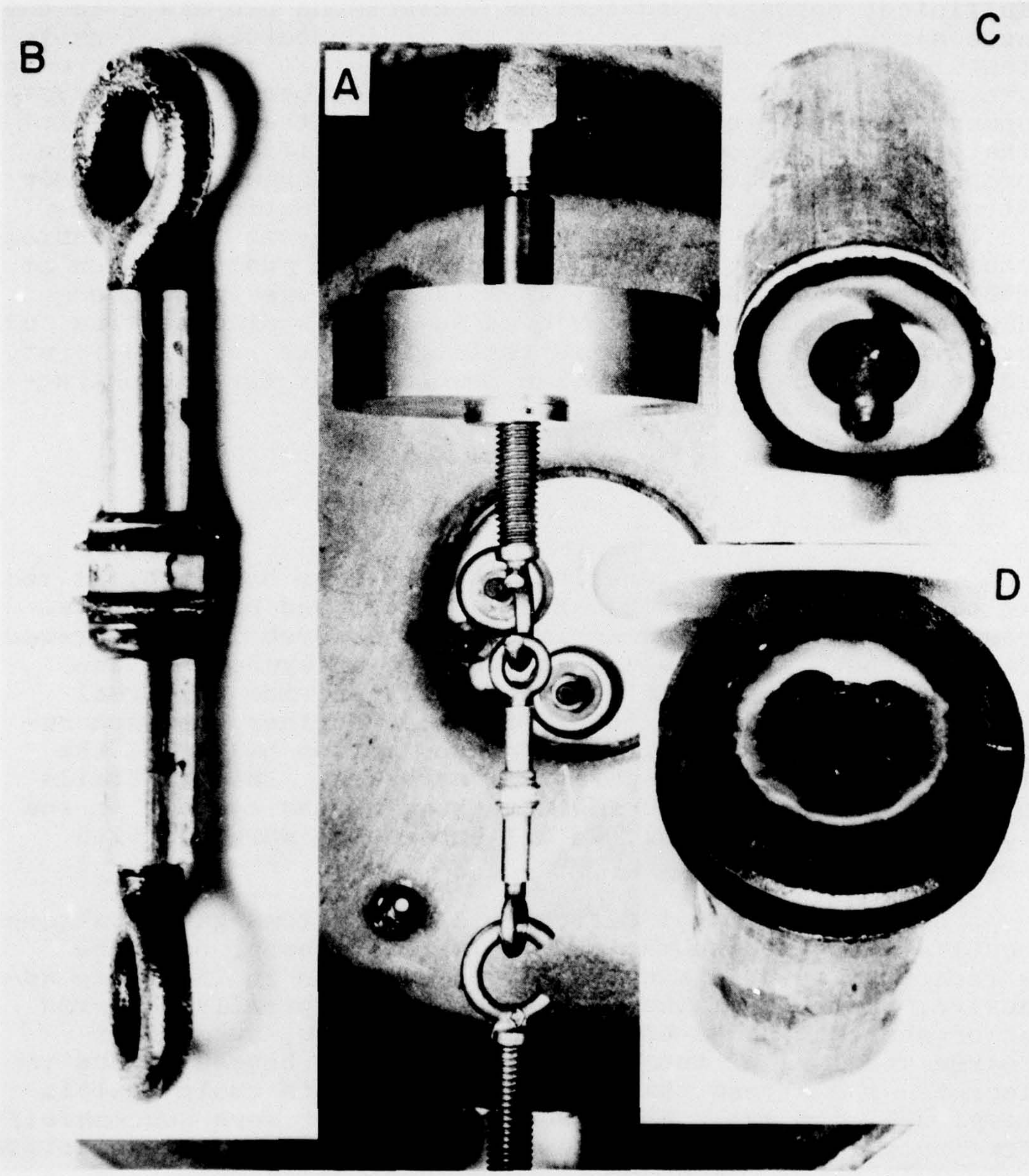

Fig. 2 — NRL tensile test of A-2 devices. (A) Device mounted in test machine. (B) Device bonded to brass attachments with threaded eye bolts. (C) and (D) Typical fracture halves of a G.E. device (No. 1567, tensile failure stress ~ 500 psi listed as a bad device).

TABLE 1

Tensile Tests of A-2 Tubes

No.	Tube: *Designation*	Load (kg)	Maximum Tensile Stress (psi) in: Seal[1]	Maximum Tensile Stress (psi) in: *Adhesive*	Failure Location
6075	Mictronic (med. good)	52	3400*	960	Adhesive
6071	Mictronic (med. good)	60	3900*	1110	Adhesive
1613	G. E. (bad)	62	3700[2]	700	Seal
1567[3]	G. E. (bad)	18	1080	200	Seal
5576	G. E. (very good)	100	6000	1120	No failure
5783	G. E. (very good)				Seal

*Estimated stress, assuming a wall thickness of 0.06".

[1] Stress in tube wall and wall seals.

[2] Stress in end seal where fracture occurred was 1364 psi.

[3] Brass grip not knurled.

a factor of 2 to 3, the problem of gripping the tubes still raised the serious question of whether sufficiently high bonding stresses could be achieved to demonstrate cracking under stress within the time and funding constraints of the program. Therefore, work on tensile testing was terminated.

Fracture and Microstructural Analysis.

Tubes that fractured in the tensile test (rather than breaking away from the brass rods) failed completely in the Al_2O_3 near the braze of the cathode (Figs. 1-3). Typically, the fracture was at or closest to the braze at the inside and outside of the braze. Such fracture is consistent with the nature and location of cracks seen by others in the Al_2O_3 near the braze as sketched in Fig. 1. The nature of the failures thus clearly indicated that they either resulted from connection of the cracks from braze stresses or were aided in propagating by these stresses. To further explore this, one tube that had a hole drilled through the cathode was first filled with red ink, then emptied prior to bonding of the brass rods and subsequent tensile testing. The tube fractured in the Al_2O_3, but at a high ($\sim$ 4,300 psi) stress. Penetration of the red ink along portions of the fracture shows that there were some pre-existing cracks in the Al_2O_3 near the braze (Fig. 3A).

Further study of the fracture surfaces of tensile tested A-2 devices were undertaken to determine from characteristic fracture markings, the direction of crack propagation. In this case, such markings are mainly fracture "tails" formed due to cracks typically not completing their passage through an intragranular pore on the same plane as the crack approached the pore, forming steps in the fracture on the opposite side of the pores until the crack again comes to a common plane. While Al_2O_3 is one of the more difficult materials in which to do this type of study, such fracture markings were found in a number of locations which clearly showed that some of the crack fronts or some of the possible multiple cracks that may have been connected together to form a complete failure were propagating from the inside surface toward the outside of the tube (Fig. 3). These fracture observations support the concept of a crack from the inside connecting voids to the interior.

Having shown the proper direction of crack propagation, it is next necessary to provide detailed characterization of the porosity present and to see if this is consistent with the proposed mechanisms. In order to do this, the porosity in sample Al_2O_3 wall sections was characterized by scanning electron microscopy of fracture surfaces instead of using the more common observation of polished surfaces.

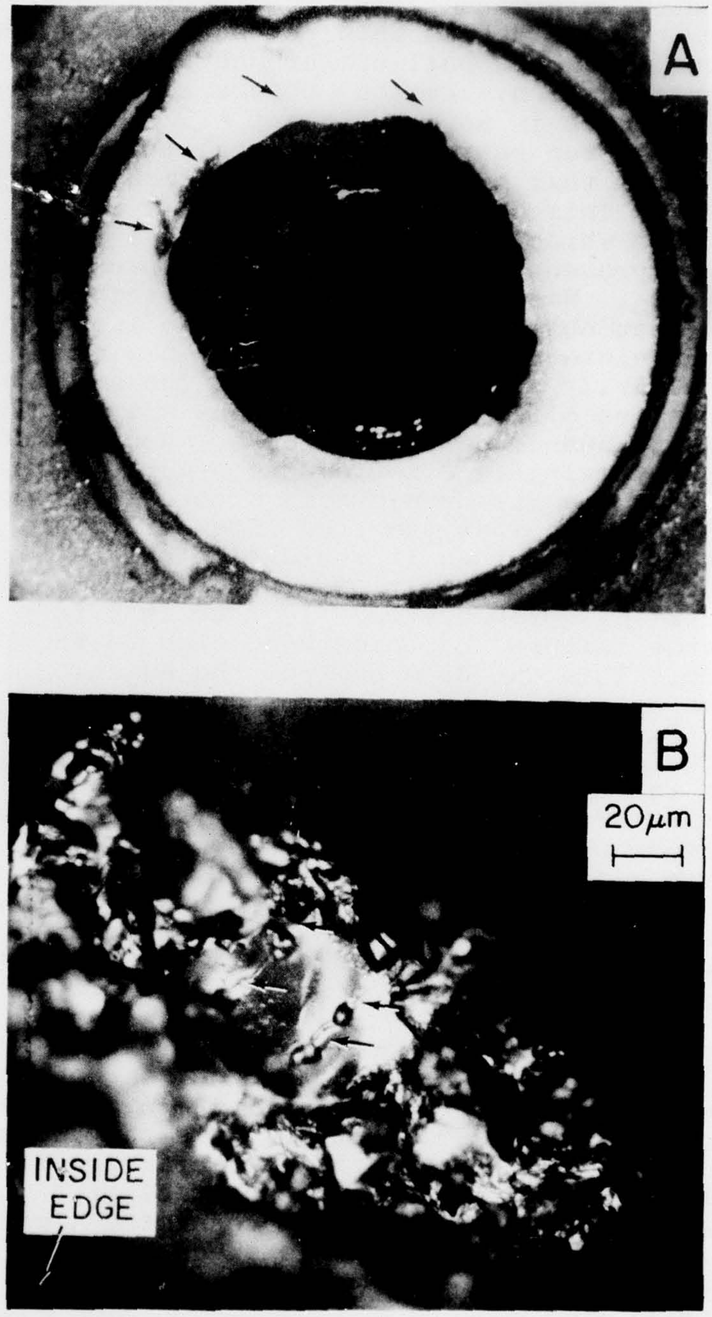

Fig. 3 — Fracture of a G.E. A-2 device. (A) Fracture of an unspecified G.E. device after dye was introduced into the device, then emptied out. Note arrows pointing to dyed areas indicating cracks in the Al_2O_3 prior to tensile failure at ~ 4300 psi. (B) Example of fracture marking (small arrows) frequently seen from pores showing crack propagation direction (large arrow).

This was done since the latter preparation often smears material or deposits debris into pores obscuring them, or preferentially causes pull-out around pores, thus preventing their identification. Further, observation of pores along fracture surfaces is more pertinent since a crack will preferentially run between pores, thus the density and character of pores that are connected by cracks is the pertinent information. This characterization shows a significant density of voids which indicates that several (Eq. 3-6) percent porosity is present as expected for a typical 96-97% commercial Al_2O_3. Basically, a bimodal distribution of pore sizes has been observed (Figs. 4-8). The larger population of pores consists of small pores about 5 microns in size located primarily at triple points in the grain boundaries. These pores are often about 20 microns apart. There is also a significant number of larger pores, about 20 microns in size, whose spacing is often of the order of 40 microns. Assuming that the gas pressure in a pore is approximately one atmosphere and that the free internal volume of the device is ~ 0.4 cm^3 then it would take only a few of the larger pores, possibly only one in the extreme to raise the internal gas pressure to $\approx 10^{-4}$ to 10^{-5} torr (assumed to be device failure) by being connected to the interior of the device. This can be seen from the equation: $n\bar{P}_p\bar{V}_p = P_T V_T$ where n = the number of pores connected to the tube interior, $\bar{P}_p$ the gas pressure in them, $\bar{V}_p$ their average volume, P_T the final tube pressure, and V_T the free internal volume of the tube. This equation indicates that of the order of 100 of the smaller voids would have to be connected to the interior of the device. This would typically require cracks from a few tens or less microns in dimensions to a few hundred microns in dimension. Thus, the virtual leak from connection of pores to the interior appears to be a viable mechanism of failure; further evidence noted below supports this hypothesis.

A key experiment supporting the pore-crack-gas leak mechanism was performed by Hass and colleagues.[3] They crushed samples of the tube wall Al_2O_3 in a vacuum in front of residual gas analyzer. Not only did they observe gas being released upon slow crushing or cracking of the Al_2O_3, but it was released in spurts, consistent with release from pores. Further, the gas released consisted almost exclusively of N_2. This is reasonable since it is well known the O_2 from air trapped in pores will readily diffuse out while N_2 will not.

Studies by Wiederhorn[4] also support the proposed mechanism. These show that thermal cycling of the tubes, similar to that which can occur in practice resulted in significant

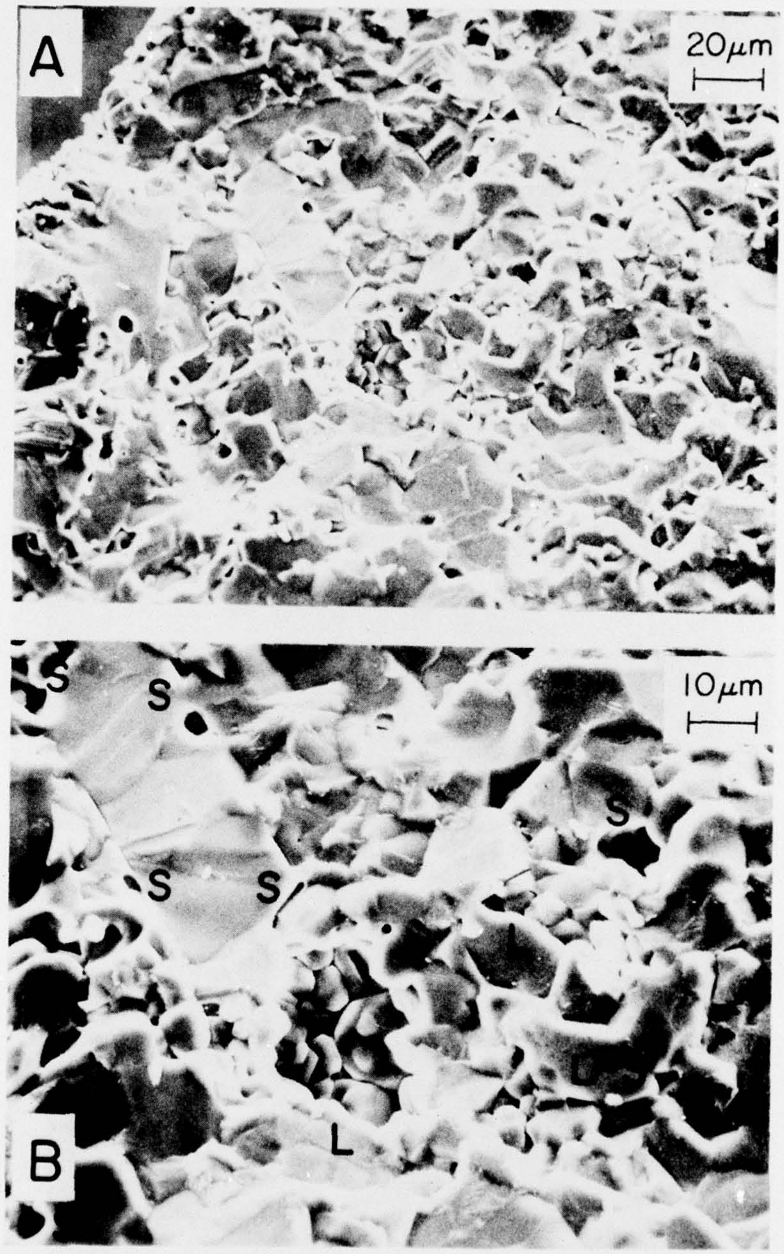

Fig. 4 — Typical microstructure of supplied A-2 Al_2O_3 fragments. (A) Lower magnification SEM micrograph. (B) Higher magnification of A. Note sample larger pores marked by L and sample smaller pores marked by S.

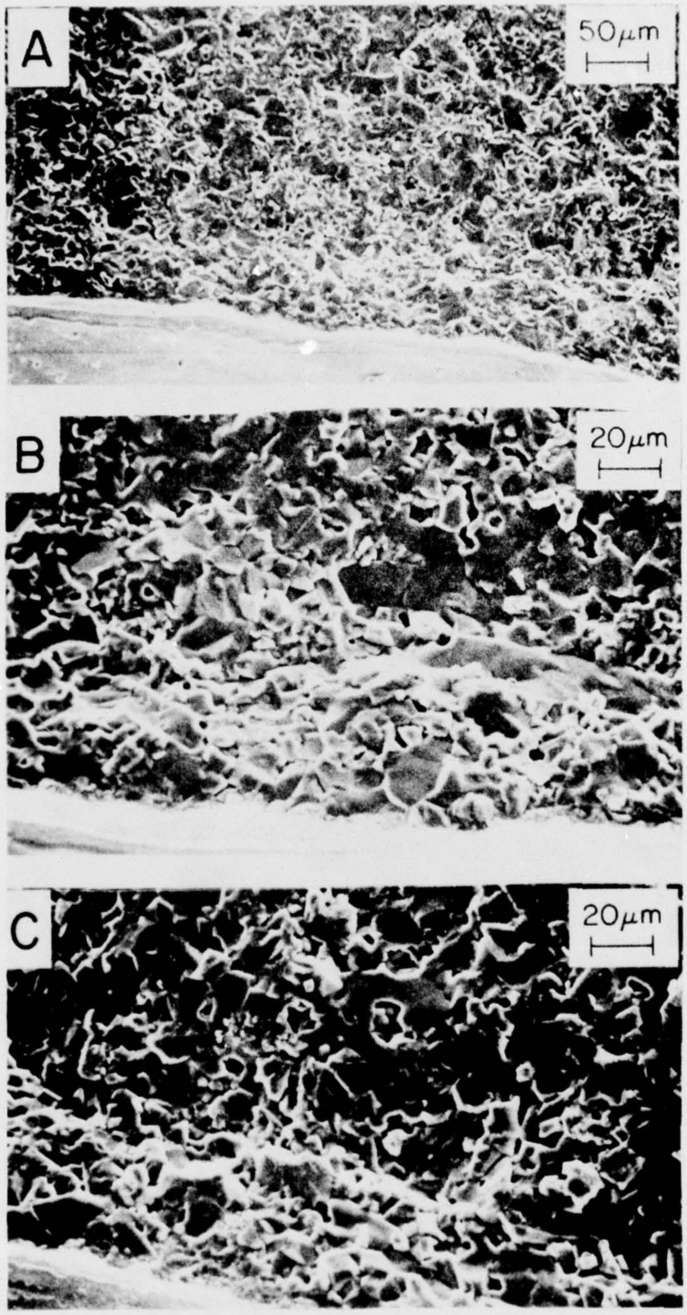

Fig. 5 — Typical microstructure near inside surface (toward bottom of (A), (B), and (C)), from G.E. A-2 No. 5783 (broken in mounting, listed as a good device).

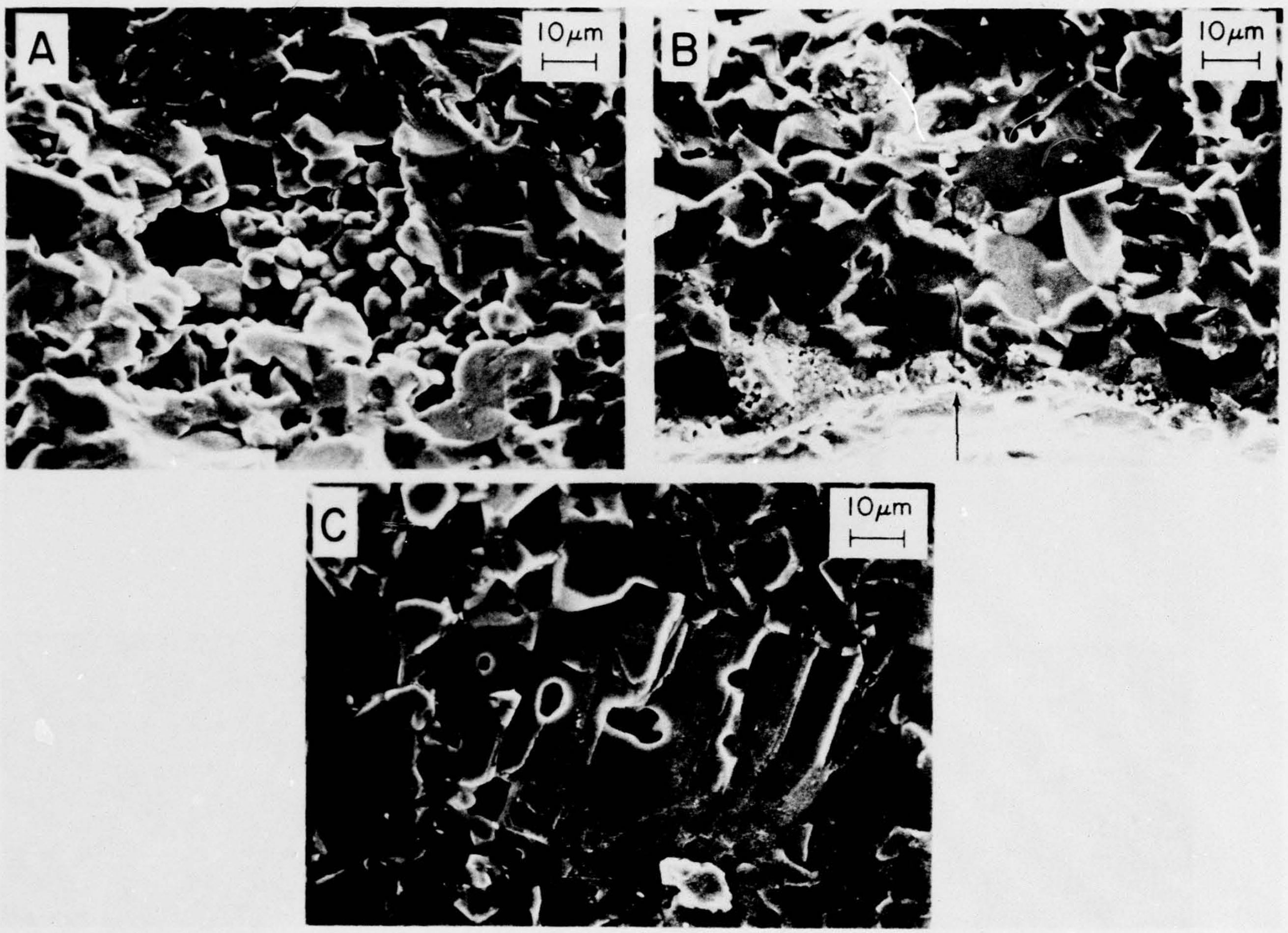

Fig. 6 — Examples of more extreme microstructural features seen on one G.E. A-2 Al_2O_3 fracture. (A) Large pore, (B) Crack propagating from the inside surface (arrow), (C) Large grain with included pores.

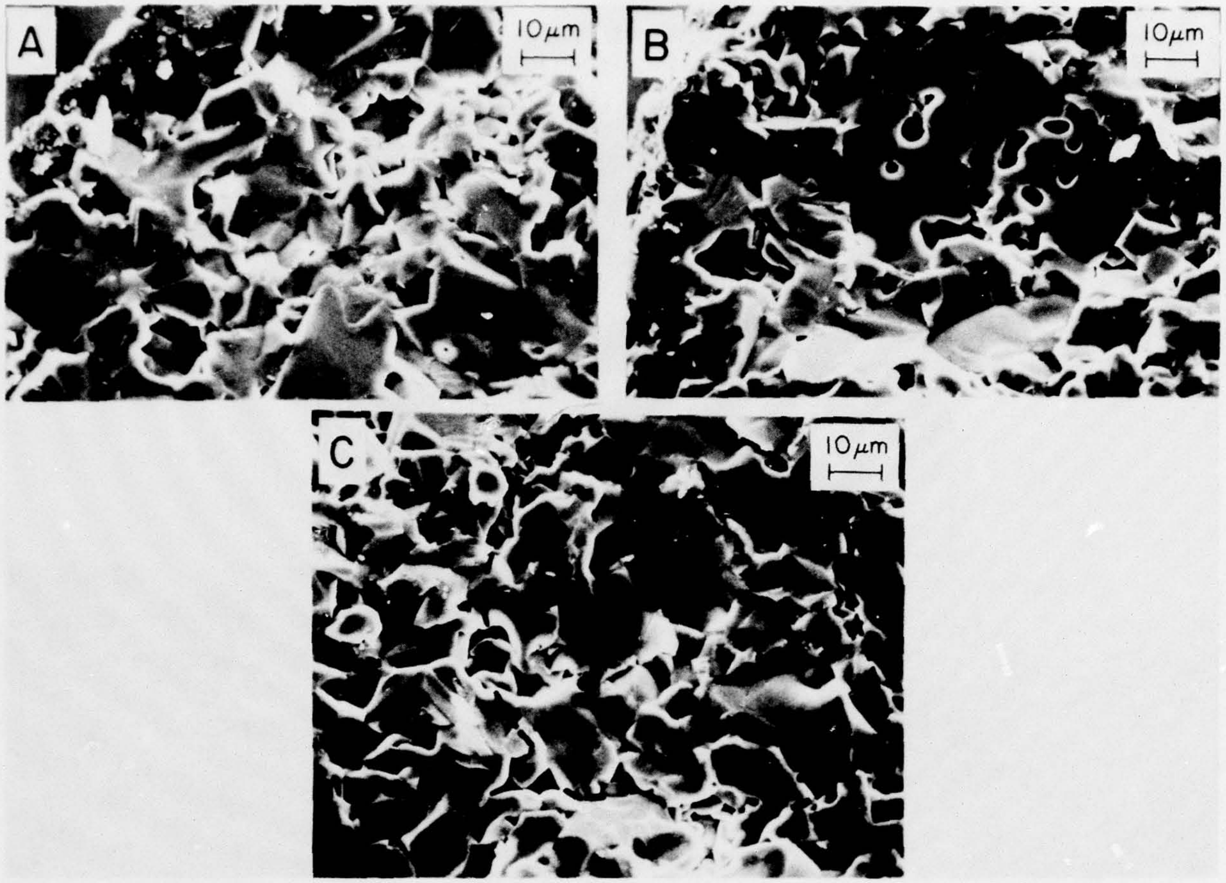

Fig. 7 — Examples of more extreme microstructural features seen on a second G.E. A-2 Al_2O_3 fracture. (A) Closely spaced larger pores, (B) Closely spaced smaller pores in larger grains, (C) Possible chain (dahsed line) of pores, e.g. around an agglomerate. All from G.E. A-2 No. 5783 (listed as a good device, broken in mounting).

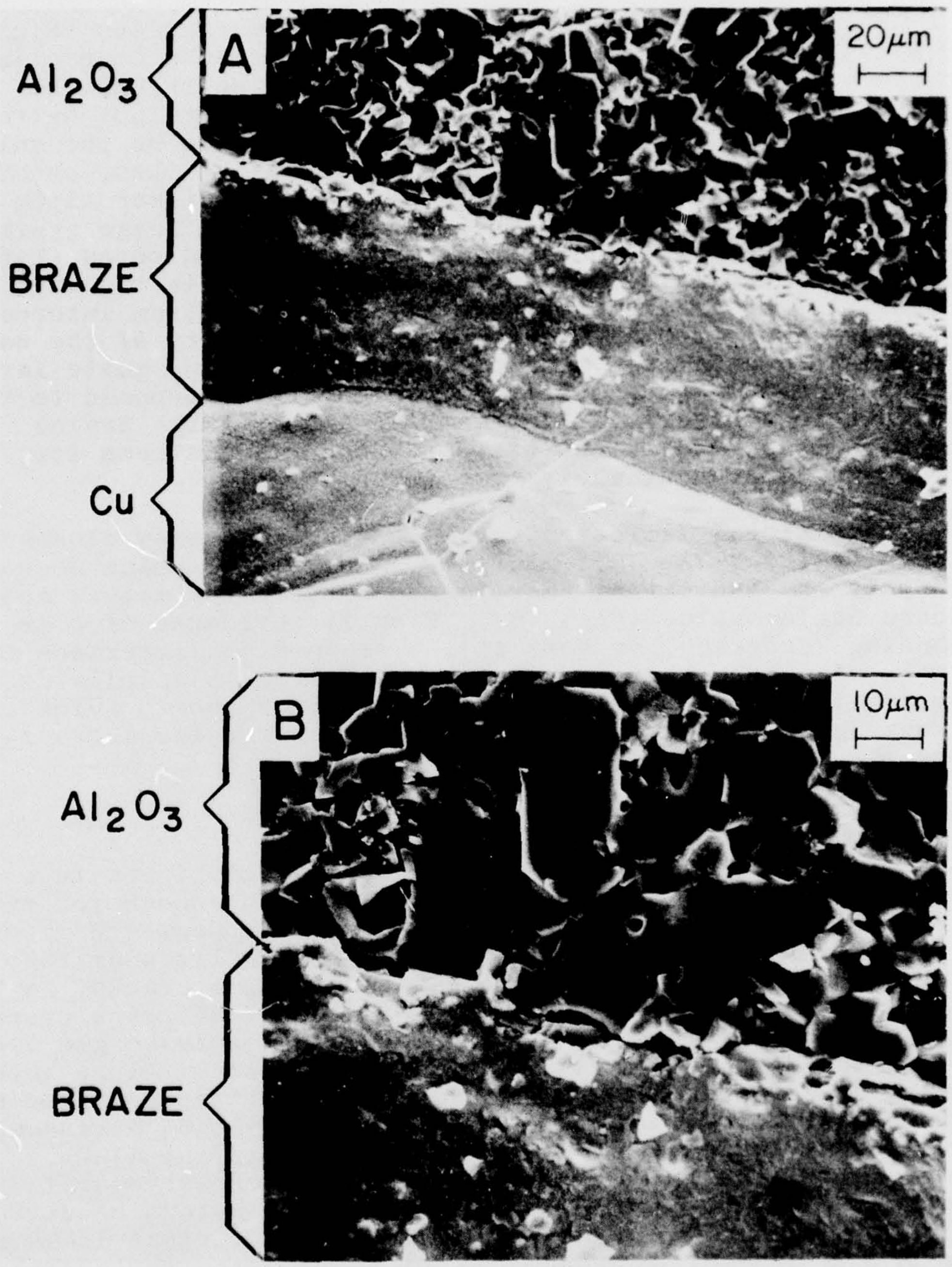

Fig. 8 — Cracked or unbonded area at the Al_2O_3-braze interface on the interior of G.E. A-2 No. 5783 (listed as a good device, broken in mounting). (B) is a higher magnification of (A).

acoustic emission, which is often indicative of crack formation or propagation.

A number of other microstructural observations (Figs. 6-8) also support the above mechanism of cracks connecting pores to provide a virtual leak. Thus, even though the microstructural observations from fracture were not extremely extensive, at least one pore about the size to be the sole cause of a virtual leak was found (Fig. 6A). Also, other occasional cracks extending outward from the inner Al_2O_3 surfaces were found (Fig. 6B). Many cases of large grains were observed (Figs. 5-7), often with included pores (Figs. 6C, 7B). Such large grains aid crack growth in two ways. First, larger grains have more strain energy from internal stresses from thermal expansion incompatibility of the non-cubic Al_2O_3 available to aid fracture. Second, quite large grains provide a low energy fracture path as opposed to the surrounding polycrystalline matrix. Thus, large grains require less, and in the extreme, no applied stress for cracking to connect their associated pores.

Additional microstructural observations show clustering of voids (Fig. 7A, 7B). Further, a number of voids observed in patterns indicating they formed between the matrix and dense agglomerates (Fig. 7C). Finally, evidence of poor bonding, cracking, or both at the braze-Al_2O_3 interface at the inside tube surface were observed (Fig. 8) similar to observations of other investigators. Such poor bonding or cracking could serve as initiating sites for the above failure mechanisms.

SUMMARY AND CONCLUSIONS

Tensile tests of A-2 devices were accomplished in a number of cases, but reaching stresses high enough for crack growth studies would require further development. However, both the existence of cracks in the Al_2O_3 walls near the brazes, the direction of propagation of these cracks, as well as the distribution of voids is such that crack propagation from the interior of the tube could release gas from voids into the tube to raise pressures to the failure levels. Cracking for such virtual leaks could result from any or all of the following: thermal stresses, braze-Al_2O_3 stresses, and thermal expansion anisotropy stresses in the Al_2O_3, especially with large grains. Further, microstructural extremes, especially large pores or pore clusters occur to aid this mechanism in some samples. Thus, virtual leaks due to cracks connecting pores and hence releasing their trapped gas to the interior is a viable mechanism of A-2 failure.

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